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POLYVINYL CHLORIDE QUALIFICATION TEST PROGRAM DEVELOPMENT AND RESULTS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

MANNED SPACECRAFT CENTER

HOUSTON, TEXAS

April 4, 1966

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POLYVINYL CHLORIDE QUALIFICATION TEST PROGRAM DEVELOPMENT AND RESULTS

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DEVELOPMENT AND RESULTS

By A. W. Wardell

SUMMARY

Polyvinyl chloride (PVC) is a polymer which had been chosen as an insulating and/or encapsulating material for certain items of Gemini government furnished equipment (GFE). Due to the inconsistencies of PVC formulations as recorded in available literature, a test program was proposed on May 10, 1965, to determine the acceptability of the selected formulations in their specific environments. The results of the test program indicated that these formulations should not be used if the ambient temperature is expected to exceed 120° F, and/or the ambient pressures will be less than 3 psia under normal or emergency conditions, as stated in Design Standards Bulletin, Serial Number DS-18, dated September 1, 1965.

Since these items of GFE were not in close proximity to ignition sources, they were not considered to be a fire hazard and flammability tests were not conducted. However, pertinent literature and prior MSC testing indicate that PVC is extremely flammable in pure oxygen.

INTRODUCTION

Gathered within this working paper will be found a complete report of all of the testing programs conducted on PVC by MSC to date. The Qualification Test Program was approved on May 14, 1965, and the procedures therein were commenced shortly thereafter. As the usage of this material was to be confined, for the present, to bioinstrumentation wiring, radiation detectors, and survival equipment, the test program included tests for tensile strength and elongation, offgassed products and weight loss in 3 psia oxygen, vacuum volatility, fungus, and odor and CO measurement.

RESUME

On April 29, 1965, a meeting was held to discuss potential problems associated with the use of polyvinyl chloride (PVC) in Gemini government-furnished equipment. As a result, a test program was prepared utilizing facilities available at the laboratories of the Structures and Mechanics Division (SMD), the Instrumentation and Electronic Systems Division (IESD), and the Crew Systems Division (CSD). The test procedures were approved by the Gemini Program Office on May 14, 1965. These procedures appear in this publication as appendix A, Qualification Test Program.

Since the test results were urgently needed prior to the Gemini IV launch, results and comments were requested on a priority basis. As PVC possesses many desirable properties, such as heat sealability and excellent flexibility characteristics, its use had been proposed in the following equipment:

a. Bioinstrumentation wiring (Calmont 3006-023-026 Type V)

b. Radiation detectors (Union Carbide "Krene", KDA-2917)

c. Survival equipment (Union Carbide, KDA-2030)

Detailed literature surveys had disclosed many seriously detrimental characteristics within the broad class of PVC polymers, among which were found:

a. Offgassing of toxic and corrosive materials

b. Migration of plasticizers to the surface, causing stickiness

c. Degradation of physical properties due to depolymerization ("unzipping") at moderate temperature/vacuum

d. Extremely high vacuum weight loss

e. Fungus nutrient characteristics

f. Objectionable odor at moderate temperatures

In considering these detrimental characteristics, the proposed tests were confined to the analyses of the following:

a. Tensile strength and elongation

b. Offgassed products and weight loss in 3 psia oxygen

c. Vacuum volatility

d. Fungus

e. Odor and CO measurement

In referring to appendix A, the Qualification Test Program in its entirety may be examined. The above list is arranged to coincide with the subdivisions of appendix A.

In adherence to the order of the proposed tests as listed above, the balance of this working paper contains summaries of the reports from the related laboratories.

Tensile Strength and Elongation

On August 2, 1965, the Experimental Structures Section reported that specimens of three different PVC materials were tensile tested between May 21 and June 23, 1965, to measure tensile strength and elongation following exposure to various environments.

Control specimens of each material were tested to establish the basic material tensile strength and elongation. Similar specimens which had been subjected to fungus exposure for 3 days and for 21 days and specimens subjected to O_2 atmosphere were then tested. All specimens of one type were cut from the same roll of material. The test results are presented in tables I through IV and summarized below.

PVC-Krene.- The control samples of Krene were cut from 0.011-inch-thick material (nominal 10 mil sheet) in both the with-grain and cross-grain directions. Samples 1 through 4A (table I) were cut with the material grain, and had an average elongation, in a 6-inch gage length, of 19.1 inches and an average strength of 23.4 pounds. Samples 5 through 9 (table I) were cut cross grain and had an average elongation of 18.3 inches and an average strength of 19.5 pounds.

All of the exposed specimens appeared to be cut in the cross-grain direction, so comparisons should be made with latter values.

The samples exposed to fungus for 3 days had been poorly prepared, and four of the seven contained edge notches or burrs which caused premature failure; only two specimens actually yielded valid test results. These results varied widely in both strength and elongation properties, so the validity of averaging the two is questionable.

PVC waterbag material.- The PVC water bag material specimens were all cut cross grain. Test results are given in table II. Specimen 3 of the specimens exposed to fungus for 3 days was well covered with fungus in several areas, but broke in an area with no fungus.

PVC wire insulation.- The PVC wire insulation specimens pulled consistently with the exception of specimen 3 of the specimens exposed to fungus for 3 days, and specimen 1 of the specimens exposed to fungus for 21 days (table III). The two specimens were damaged in stripping the insulation from the wire. A summary of the test results is given in table IV.

The results of the fungus tests on the Krene and waterbag material were rather inconclusive as the data were somewhat scattered. There appeared to be no significant change after 3-day exposure to fungus. There was a slight loss of elongation and strength after 21 days of exposure. The wire insulation tests, however, were much more consistent and indicate just the reverse, with both elongation and strength being slightly higher after the 21 days of exposure.

The tests indicate that O_2 exposure had no discernable effect on the tensile strength or elongation of the PVC materials (table IX).

Mechanical properties following thermal/vacuum exposure.- The report dated July 9, 1965 disclosed the following results of the thermal/vacuum tests on polyvinyl chloride (PVC):

The Mechanical Systems Laboratory (MSL) performed a series of thermal/vacuum tests on polyvinyl chloride (PVC) materials to determine if there were possible problems associated with its use in Gemini government-furnished equipment. Four samples of Union Carbide KDA-2030 were tensile tested after a 4-day thermal/vacuum exposure in the turbomolecular pumping system (TMP) of the MSL. Two samples of Union Carbide "Krene" KDA-2917 were tensile tested after a 3-day thermal/vacuum exposure in the Ainsworth vacuum balance of the Spacecraft Materials Laboratory. Both materials showed significant changes in the tensile test properties as a result of these exposures. However, the performance of both of these materials may not be seriously impaired in their spacecraft application. The "Krene" material is used in radiation detectors inside the astronaut's spacesuit and, therefore, will not be exposed to vacuum or high temperature. The KDA-2030 which is used in the survival equipment could be exposed to vacuum and high temperatures, but certainly less severe than in these tests. The elongation is the property most affected by the thermal/vacuum exposure, but even so, the material could not be described as brittle, having an elongation of well over 100 percent after the 4-day, 160° F thermal/vacuum exposure.

Testing was done in apparatus designed for thermal/vacuum tests of parachute materials. The TMP has six individual sample chambers with the capability of performing a tensile test inside each chamber, either while at the vacuum condition or after a controlled repressurization. Heating of the sample can be done while under vacuum, and analysis of outgassing products can be done at any time during the vacuum exposure. Pull force is provided by an aircraft linear actuator, and measured by a strain-gage type load cell. Elongation is measured by a linear potentiometer, and test data is recorded on a recording oscillograph. A frame is provided for control tests which can utilize the same test hardware and instrumentation that is used for the thermal/vacuum samples. Sample holders used are flat-faced grips made of stainless steel, with cotton webbing on both sides of the test sample to protect it from holder damage.

Two test samples of "Krene" were cut from a piece of material that had been used for a vacuum weight loss test, and tensile tested in the control test frame. These samples had a 3-day vacuum exposure (pressure about 1×10^{-6} torr) at 160° F, and showed considerable stiffening to the touch from the original condition. The primary change, resulting from the thermal/vacuum exposure, was the loss of elasticity due to loss of the plasticizer. Ultimate strength levels were not degraded from the original condition. Test values are shown in table V.

Ultimate elongation values on the "Krene" control sample and the KDA-2030 control sample are estimated, since in order to break the sample, it had to be remounted in the holders after the first pull. The stroke available from the linear actuator is limited to about 4 inches, and 1.5 inches is the shortest sample length that can be tested.

The Union Carbide KDA-2030 samples were mounted in tensile test holders inside the thermal/vacuum chambers so that the samples could be tested without removing them from the chambers. Two samples (chambers 2 and 5) were started on May 18, 1965, and exposed to vacuum and 160° F for 4 days. A second pair of samples (chambers 1 and 3) were started on May 21, 1965, and exposed to vacuum for 4 days - 1 day at 160° F and 3 days at 120° F. The pressure level was not monitored in the test chamber for the first pair of samples; however, pressure readings were taken at the base plate which was equivalent to the chamber exit pressure. The pressure was monitored on Chamber 1 of the second pair of samples. These measurements indicated that after the initial few hours of pumping on the test chamber, a pressure differential of about 2.5 decades was maintained between the test chamber and the test chamber exit. The first sample pair, therefore, was exposed to a pressure level of about 5×10^{-6} torr. The gas load from the second pair was less because of the lower exposure temperature and, therefore, resulted in a slightly lower pressure in the sample chamber of about 3×10^{-6} torr. The gas load from the samples was high throughout the 4 days on both sets of samples and showed no indication of falling off at the end of the 4 days. By comparison, the same size piece of 300 lb nylon tape causes about a 1 decade pressure differential between the test chamber and the chamber exit.

Inspection of chambers 2 and 5, after removal of the KDA-2030 samples, showed that some of the outgassing products were condensable materials, in that some of the cooler surfaces in the chambers were stained with a thin coating of material. The first attempt to test the KDA-2030, after a 4-day thermal/vacuum exposure (chamber 5 in table VI), was made while the material was still at 160° F and in the vacuum. When the sample did not break, the heat was turned off, and the sample repressurized with ambient air in order to speed cooling; however, the sample was left in the chamber. Another attempt, after the sample had cooled to room temperature, also failed to break the sample. Later inspection of the specimen showed that much of the elongation occurred in the material right at the holders which had been somewhat protected from the vacuum exposure by the cotton webbing. The configuration of the test chambers limits the stroke of the holders, while in the chamber, to about 3.5 inches.

The Chamber 2 sample was allowed to cool, and was repressurized before being tested. Again the sample did not break. The sample was then removed from the test chamber, and visual inspection again showed that the material, right at the holders, was doing most of the elongation. The center portion of the sample was then successfully tested in the control frame.

The second pair of KDA-2030 samples was tested by removing them from the thermal/vacuum chambers, and testing the center 1.5 inches of the 2 inches of original sample length (sample length is equivalent to length of exposed sample between holders).

The test results are definitely influenced by the loading rate as evidenced by the rapid relaxation of tensile load when the test sample was held at a given elongation. Tensile values may also be influenced by previous stress history, as in the case where samples were pulled twice before breaking.

Test data for the KDA-2030 are summarized in table VI.

The final thermal/vacuum tests, reported on October 27, 1965, were conducted to determine the effects of a thermal/vacuum environment on

PVC as a wire insulation material. Four samples were tested under ambient conditions in an Instron tensile test machine, and the breaking strength and elongation recorded. The breaking strength varied from 6.1 pounds to 6.4 pounds, and the elongation varied from 207.5 percent to 227.5 percent. Four samples were exposed to a vacuum of 1×10^{-6} mm Hg and a temperature of 160° F for 4 days. These samples were returned to ambient temperature and pressure, and then tensile tested in the Instron test machine. The breaking strength varied from 6.45 pounds to 6.9 pounds, and the elongation varied from 175 percent to 225 percent.

As is evidenced by the data, it is concluded that no appreciable strength or elongation change resulted from the thermal/vacuum exposure. For comparison purposes, table VII shows the results of the tests on unexposed samples, and table VIII reveals the results on exposed specimens.

Offgassed Products and Weight Loss in 3-psia Oxygen

The following report, dated June 17, 1965, discloses the results of the outgassing investigation of polyvinyl chloride (PVC) film, as performed by the Analytical Instrumentation Calibration Laboratory:

Outgassing analysis of several polyvinyl chloride materials, to determine suitability for spacecraft use, was begun on May 7, 1965. The following materials were tested during the indicated intervals:

May 7, 1965 through May 11, 1965: "Krene" KDA 2917 (PVC film)

May 13, 1965 through May 17, 1965: Calmont wire 3006-023-26, type V

May 21, 1965 through May 25, 1965" "Krene" KDA 2030 (PVC film)

Test results were as follows:

"Krene" KDA 2917.-

a. Two samples, each 2 feet by 2 feet, cut and weighed. Weights were:

No. 1 – 142.1584 grams

No. 2 – 142.9061 grams

b. Chamber evacuated and purging started at 2:45 p.m., May 7, 1965.

c. Background sample of purge gas taken at 3:45 p.m., May 7, 1965. This sample was analyzed on a Beckman IR-9 infrared spectrophotometer. The absorption spectrum showed CO_2 and H_2O vapor which are normal components of breathing grade oxygen used in this test.

d. Test started at 4:00 p.m., May 7, 1965.

e. Sample No. 1 taken at 6:00 p.m., May 7, 1965. This sample was analyzed on the IR-9 spectrophotometer. The spectrum showed only CO_2 and H_2O vapor.

f. Test continued for 4 days at 120° F and 3 psia of 100 percent O_2.

g. Sample No. 2 taken at 4:00 p.m., May 11, 1965, and analyzed on IR-9. Only CO_2 and H_2O vapor were found.

h. Samples of film reweighed on May 11, 1965.

No. 1 – 142.1338 grams

No. 2 – 142.8795 grams

Weight loss was:

No. 1 – 0.0246 grams

No. 2 – 0.0266 grams

or approximately .02 percent weight loss

Calmont wire 3006-023-26, type V.-

a. A 1-foot piece of wire was stripped and the insulation weighed. Weight was 0.7740 gram.

b. A 30-foot piece of wire was weighed complete. Weight was 50.1049 grams. This sample was used in the test.

c. Sample No. 3 was taken for background after a 1-hour purge, with 100 percent O_2 at ambient temperature.

d. Test started at 2:00 p.m., May 13, 1965.

e. Sample No. 4 was taken at 3:15 p.m., May 13, 1965. Sample analyzed on IR-9. Only CO_2 and H_2O vapor were detected.

f. Test continued for 4 days at 120° F and 3 psia of 100 percent O_2.

g. Sample No. 5 was taken at 2:00 p.m., May 17, 1965, and analyzed on IR-9. Only CO_2 and H_2O vapor were detected.

h. Wire (b) was reweighed on May 17, 1965. Weight was 50.0540 grams. Weight loss was 0.0509 gram or approximately 0.1 percent.

Union Carbide, KDA-2030.-

a. Two pieces, each 18 inches by 36 inches, were prepared and weighed on May 21, 1965.

No. 1 – 125.8932 grams

No. 2 – 131.9058 grams

b. Sample No. 6 was taken at 5:45 p.m., on May 21, 1965, after a 1-hour purge with 100 percent O_2 at ambient temperature. This was a reference sample and showed only the presence of CO_2 and H_2O vapor.

c. Test started at 6:30 p.m., May 21, 1965.

d. Sample No. 7 was taken at 7:45 p.m., May 21, 1965. Only CO_2 and H_2O vapor were detected.

e. Test continued for 3 days and 23 hours at 120° F and 5.5 psia of 100 percent O_2. Last hour of the 4-day test was at 160° F and 5.5 psia of 100 percent O_2.

f. Sample No. 8 was taken at 6:40 p.m., May 25, 1965, and analyzed on IR-9. This sample was analyzed at a 10-meter path length. The components identified were CO_2, H_2O vapor, C_2H_2 (acetylene, a normal component of breathing grade oxygen), and a trace of what is probably freon (source unknown).

g. Film samples were reweighed on May 25, 1965.

No. 1 – 125.6150 grams

No. 2 – 131.7500 grams

Weight loss was:

No. 1 – .2787 gram

No. 2 – .1557 gram

or approximately .17 percent

Vacuum Volatility

In the report from the Spacecraft Materials Section, dated July 16, 1965, the subject of weight loss of some PVC used in GT-4 was discussed and the following report given:

The water bag and radiation badge packets used on GT-4 were made of PVC. Studies were made in order to determine if this material is suitable for use in vacuum environments. One of these studies was weight loss in vacuum environments, and was rigidly conducted.

Samples of water bag sheet (Union Carbide KDA 2030) 0.01 inch thick, and radiation packet sheet 0.012 inch thick, were supplied by the Crew Systems Division. Suitable size samples were cut from these sheets and weighed. They were then inserted in an Ainsworth Vacuum Balance, which is capable of recording continuous weight changes at pressures of approximately 10^{-6} torr. The sample temperature was raised to 71° C, and weight loss was observed for approximately 100 hours. The tests were terminated and the samples were visually inspected.

Figure 1 shows the weight loss history for one sample of radiation packet material and two samples of water bag material. Surface area and sample weight are indicated in the legend. The total weight loss after 96 hours is quite large for both (17.7 percent for the radiation packet polymer, and 10.5 percent for the water bag polymer). Agreement between the two samples of water bag material is good.

Visual examination of the sample after testing revealed a considerable change in flexibility of both materials.

The major outgassing product condensed on the balance walls as a clear, viscous liquid. No attempts were made to identify this component; however, earlier studies with Tygon tubing, which is a PVC, indicated this material to be a plasticizing agent.

One can conclude from the data mentioned that the materials were both degraded at a temperature of 71° C, and a pressure of 10^{-6} torr.

Further, the outgassing product could possibly lead to contamination of other spacecraft components.

In a later report, dated July 21, 1965, it was disclosed and recommended that the use of the three PVC formulations tested be prohibited for applications which would be exposed to greater than 120° F, and/or less than 3 psia. The final results of these tests, from which the above conclusion was derived, appear in table IX entitled "Summary of Results."

Fungus

The results of the fungus resistance tests on PVC were reported by the Crew Systems Division on July 21, 1965. Three formulations of PVC were tested for resistance to mycoidal growth. The materials tested were:

a. Wire – Calmont 3006-023-026 type V (bioinstrumentation wiring)

b. Sheet – Union Carbide "Krene" KDA 2917 (radiation detectors)
Union Carbide KDA 2030 (Survival equipment)

For testing purposes, the sheet material was cut into strips $\frac{1}{2}$ inch wide and 12 inches long, and the wire sample was cut into 12-inch lengths. The test samples were gently wiped to remove dust, but were not sterilized.

The test culture was composed of bacterial species indigenous to the human skin, and mycoidal species found as common air contaminants. This culture was swabbed over the surface of Sabouraud's Dextrose Agar contained in Petri dishes prior to the placement of the test samples. One piece of each type of polyvinyl chloride was placed against uninnoculated agar, and six against innoculated agar in flattened spirals, with as much of the sample against the agar as possible. The wire samples were loosely coiled, and placed against the agar. The control samples and five of the test samples were incubated at 37° C. The remaining test samples were incubated in the dark at ambient temperature.

The plates were examined at intervals during the test period. At the end of 14 days, the fungus had attained about 80 percent of the growth evident at 21 days. On the 21st day, the plates were closely examined to determine the extent of the growth on, or adjacent to, the PVC samples. In all cases, the mycoidal growth had formed a bridge between the agar and the PVC. However, growth extending further than 1 millimeter onto the specimen was evident only as follows:

a. Wire – The control and four of the samples incubated at 37° C were completely ringed with growth in one or more areas.

b. "Krene" KDA 2917 – The strips showed minimal growth extending 1 millimeter or more onto the strips; however, the dosimeter packets made of this material showed rather luxuriant growth extending as far as the center of the packet.

c. KDA 2030 – All samples showed growth extending at least 1 millimeter onto the strip from the edge. In addition, the five test samples incubated at 37° C showed droplet-like colonies at intervals down the long axis of the strip.

During actual spacecraft operation or test conditions, there has been no current evidence that fungal growth has occurred on these materials.

Odor and CO Measurement

Tests on PVC for odor and CO measurement were conducted in accordance with the procedures outlined in the initial Qualification Test Program. (See appendix A.) The results of the tests on radiation detectors (Union Carbide "Krene" KDA-2917), water bags (Union Carbide KDA-2030), and bioinstrumentation wire (Calmont 3006-023-026, type V), were reported as "Acceptable" on April 2 and June 10, 1965.

In referring to Odor Test and CO Measurement in appendix A, the extent of the tests conducted may be examined in detail. The rigidity of the test program will be noted; therefore, through the acknowledgment of the characteristics as "Acceptable", it may be assumed, as far as odor and CO measurements are concerned, that no seriously undesirable qualities exist in this area.

Table IX, the Summary of Results, briefly yet concisely summarizes the results of the five basic tests conducted and may be considered as conclusive.

CONCLUSIONS

All three PVC formulations tested showed unacceptable weight loss characteristics when exposed to 1×10^{-6} torr at 160° F. Two of the three formulations showed unacceptable mechanical properties following this thermal vacuum exposure. All three were fungus nutrient, although the extent of the hazard to the astronauts has not been determined.

The results of the remaining tests were considered acceptable, as outlined in table IX, Summary of Results, page 22.

TABLE I.- PVC-KRENE

Specimen no.	Width, in.	Gage length, in.	Extension (L), in.	Failure load, lb	Comments
Control specimens					
1	0.5	6.0	16.865	22.5	With material grain
2			20.740	26.6	With material grain
3			18.955	25.8	With material grain
4			18.490	22.2	With material grain
4A			20.475	19.8	With material grain
5			17.730	19.5	Cross grain
6			17.770	18.8	Cross grain
7			20.280	19.7	Cross grain
8			16.382	15.7	Cross grain
9			19.440	23.6	Cross grain
3-day fungus-exposed specimens					
1	0.5	6.0			Slipped in jaws
2			11.850	12.8	Edge notch failure
3			9.980	12.1	Edge notch failure
4			9.485	12.1	Edge notch failure
5			22.940	22.2	Clean break
6			9.718	11.2	Edge notch failure
7			17.100	17.7	Clean break
21-day fungus-exposed specimens					
1	0.5	6.0	20.270	19.7	Clean break
2			17.515	18.2	Edge notch failure
3			16.725	19.2	Clean break
4					Slipped in jaws
5			18.480	22.5	Clean break
6			12.405	16.3	Clean break - in fungus
7			6.890	11.5	Edge notch failure
O_2-exposed specimens					
1	0.5	6.0	16.770	17.8	
2			19.100	20.5	
3			19.100	20.5	

TABLE II.- PVC-WATERBAG MATERIAL

Specimen no.	Width, in.	Gage length, in.	Extension (L), in.	Failure load, lb	Comments
Control specimens					
1	0.5	6.0	16.870	20.5	
2			15.220	19.0	
3			15.450	19.0	
3-day fungus-exposed specimens					
1	0.5	6.0	6.618	11.0	Edge notch failure
2			16.670	18.3	Clean break
3			16.950	18.7	Clean break
4			13.013	15.5	Edge notch failure
5			17.850	18.1	Clean break
6			15.040	17.2	Edge notch failure
7			17.910	21.5	Clean break
21-day fungus-exposed specimens					
1	0.5	6.0	9.775	17.7	Edge notch failure
2			15.480	19.3	Clean break
3			14.895	18.4	Clean break
4			10.270	14.2	Clean break
5			16.800	17.4	Clean break
6			18.135	19.7	Clean break
7			11.725	14.6	Clean break
O_2-exposed specimens					
1	0.5	6.0	16.770	20.5	
2			12.670	17.5	
3			16.740	19.8	
4			17.100	20.8	

TABLE III.- PVC-WIRE INSULATION

Specimen no.	gage length, in.	Extension (L), in.	Failure load, lb	Comments
Control specimens				
1	6.0	14.965	6.4	
2		14.955	6.2	
3				Slipped in grips
4		16.925	6.6	
5		16.950	6.6	
6		17.735	6.9	
7		15.895	6.6	
3-day fungus-exposed specimens				
1	6.0			Slipped in jaws
2		15.650	6.6	
3		12.125	5.6	Insulation damaged in stripping
4		16.625	6.9	
5		13.475	6.3	
6		16.610	7.1	
7		15.805	6.8	
21-day fungus-exposed specimens				
1	6.0	14.985	7.1	Insulation damaged in stripping
2		18.040	7.5	
3		17.340	6.8	
4		17.635	7.1	
5		16.520	6.7	
6		17.740	6.9	
7		16.170	6.7	
O_2-exposed specimens				
1	6.0	14.825	6.5	
2		16.855	7.1	
3		14.210	6.5	
4		16.740	6.9	
5		16.300	6.8	
6		16.500	6.7	
7		16.885	6.7	

TABLE IV.- SUMMARY OF AVERAGE VALUES OF

ELONGATION AND STRENGTH OF PVC SPECIMENS

Condition of specimen	Average elongation, in.	Average strength, lb
Krene		
Control	18.3	19.5
[a]3-day Fungus	20.0	19.9
21-day Fungus	17.0	19.4
O_2 Atmosphere	18.3	19.6
Waterbag material		
Control	15.9	19.5
3-day Fungus	17.3	19.1
21-day Fungus	14.6	17.3
O_2 Atmosphere	15.8	19.6
Wire insulation		
Control	16.2	6.5
3-day Fungus	15.1	6.7
21-day Fungus	17.2	6.9
O_2 Atmosphere	16.0	6.7

[a]Average of only two samples.

TABLE V.- PVC THERMAL VACUUM TESTS

UNION CARBIDE "KRENE" KDA-2917. TESTED 13 MAY 1965, MECHANICAL SYSTEMS LABORATORY

[Tested at ambient temperature and pressure loading speed 1.5 in. sample at 26 IPM]

Sample description	Vacuum exposure	Elongation, percent	Max. load, lb/in	Comments
Control	None	300+	43.0	Sample had to be remounted in order to break; elongation is estimated.
No. 1	3 days at 160° F 1×10^{-6} torr	(5) 86.7	(53.3) 44.0	Maximum load did not occur at maximum elongation.
No. 2	3 days at 160° F 1×10^{-6} torr	(5) 77	(55.9) 45.0	Same as sample No. 1

TABLE VI.- PVC THERMAL VACUUM TESTS

UNION CARBIDE KA-2030, MECHANICAL SYSTEMS LABORATORY, MAY 1965

[All tensile tests done at 26 IPM]

Sample description	Date	Vacuum exposure	Test pressure	Test temperature, °F	Sample length, in.	Elongation, percent	Maximum load, lb/in.	Break	Comments
Control	May 17	None	Ambient	Ambient	1.5	300+	41.6	Yes	Sample had to be remounted to break; elongation estimated.
Chamber 5	May 22	4 days at 160° F 5×10^{-6} torr	Vacuum Ambient	160 86	2.0 2.0	171 171	21.6 16.1	No No	Relaxed to 16.8 lb in 7 sec. Stretched at holder
Chamber 2	May 22	4 days at 160° F 5×10^{-6} torr	Ambient Ambient	80 Ambient	2.0 1.5	169 (40) 127	30.0 (40.8) 31.5	No Yes	Stretched at holder. Maximum load not at maximum elongation; remounted sample in control frame.
Chamber 1	May 25	4 days 1 at 160° F 3 at 120° F 3×10^{-6} torr	Ambient	Ambient	1.5	(37) 250	(30.4) 31.3	No	Tested sample in control frame; load relaxed to 17 lb in 5 sec.
Chamber 3	May 25	4 days 1 at 160° F 3 at 120° F 3×10^{-6} torr	Ambient	Ambient	1.5	220	31.8	Yes	Tested sample in control frame.

TABLE VII.- PVC THERMAL VACUUM TESTS

CALMONT 3006-023-026, TYPE V, WIRING INSULATION

UNEXPOSED SAMPLES

Sample No. 1	Breaking strength	Elongation information
1	6.10 lb	23.34 in. - jaw travel From 1 in. to 3.20 in. or $\frac{3.20-1 \times 100}{1} = 220$ percent
2	6.25 lb	26.32 in. - jaw travel From 1 in. to 3.25 in. or $\frac{3.25-1 \times 100}{1} = 225$ percent
3	5.90 lb	23.60 in. - jaw travel From 2 in. to 6.15 in. or $\frac{6.15-2 \times 110}{2} = 207.5$ percent
4	6.40 lb	25.08 in. - jaw travel From 2 in. to 6.55 in. or $\frac{6.55-2 \times 100}{2} = 227.5$ percent

TABLE VIII.- PVC THERMAL VACUUM TESTS

CALMONT 3006-023-026, TYPE V, WIRING INSULATION

EXPOSED SPECIMENS

[1×10^{-6} mm Hg 160° F - Four days exposure]

Sample No. 1	Breaking strength	Elongation information
1	6.45 lb	18.88 in. - jaw travel 2 in. to 5.5 in. or $\frac{3.5}{2}$ = 175 percent
2	6.70 lb	18.60 in. - jaw travel 2 in. to 6.3 in. or $\frac{4.3}{2}$ = 215 percent
3	6.90 lb	20.44 in. - jaw travel 2 in. to 6.5 in. or $\frac{4.5}{2}$ = 225 percent
4	6.90 lb	19.12 in. - jaw travel 2 in. to 6.3 in. or $\frac{4.3}{2}$ = 215 percent

TABLE IX.- SUMMARY OF RESULTS

Test	Acceptance criteria	Measured values		
		Bioinstrumentation wire (Calmont 3006-023-026, Type V)	Radiation detectors (Union Carbide "Krene", KDA-2917)	Water bags (Union Carbide KDA-2030)
A. Mechanical properties after following tests:				
1. Thermal vacuum exposure (Test C)				
a. Tensile test	30 percent maximum allowable decrease	9.4 percent increase	No change	61.4 percent decrease[a]
b. Elongation test	30 percent maximum allowable decrease	5.5 percent decrease	74.4 percent decrease[a]	57.7 percent decrease[a]
2. Fungus (Test D)				
a. Tensile test	30 percent maximum allowable decrease	6 percent increase	No change	11.3 percent decrease
b. Elongation test	30 percent maximum allowable decrease	15 percent increase	19 percent decrease	22 percent decrease
3. 3 psia O_2 (Test B)				
a. Tensile test	30 percent maximum allowable decrease	3 percent increase	No change	No change
b. Elongation test	30 percent maximum allowable decrease	4 percent decrease	No change	No change
B. Offgassed products and weight loss in 3 psia O_2 at 120° F	a. CSD analysis for toxic products	Acceptable (CO_2 and H_2O)	Acceptable (CO_2 and H_2O)	Acceptable (CO_2 and H_2O, C_2H_2, "Freon")
	b. 2.0 percent weight loss for first 24 hours	0.2 percent weight loss in 96 hours	0.02 percent weight loss in 96 hours	0.17 percent weight loss in 96 hours
C. Vacuum volatility (160° F, 1×10^{-6} torr)	a. Stable rate of weight loss not greater than 0.0025 percent/hr	0.016 percent[a]	Did not stabilize[a]	Did not stabilize[a]
	b. 2.0 percent weight loss for first 24 hours	2.1 percent in first 24 hours; 3.5 percent in 96 hours[a]	12.35 percent in first 24 hours; 17.7 percent in 96 hours[a]	6.44 percent in first 24 hours; 10.5 percent in 96 hours[a]
D. Fungus (100° F, 95 percent ± 5 percent R.H.)	No bacteriological/mobility hazard to suited astronaut	Fungus nutrient Hazard not determined	Fungus nutrient Hazard not determined	Fungus nutrient Hazard not determined
E. Odor and CO measurement	Per CSD-G-249	Acceptable per memo to Gemini Program Office from Crew Systems Div. dated June 10, 1965	Acceptable per memo to Gemini Program Office from Systems Test Br. dated April 2, 1965	Acceptable per memo to Gemini Program Office from Systems Test Br. dated April 2, 1965

[a]Indicates unacceptable characteristics

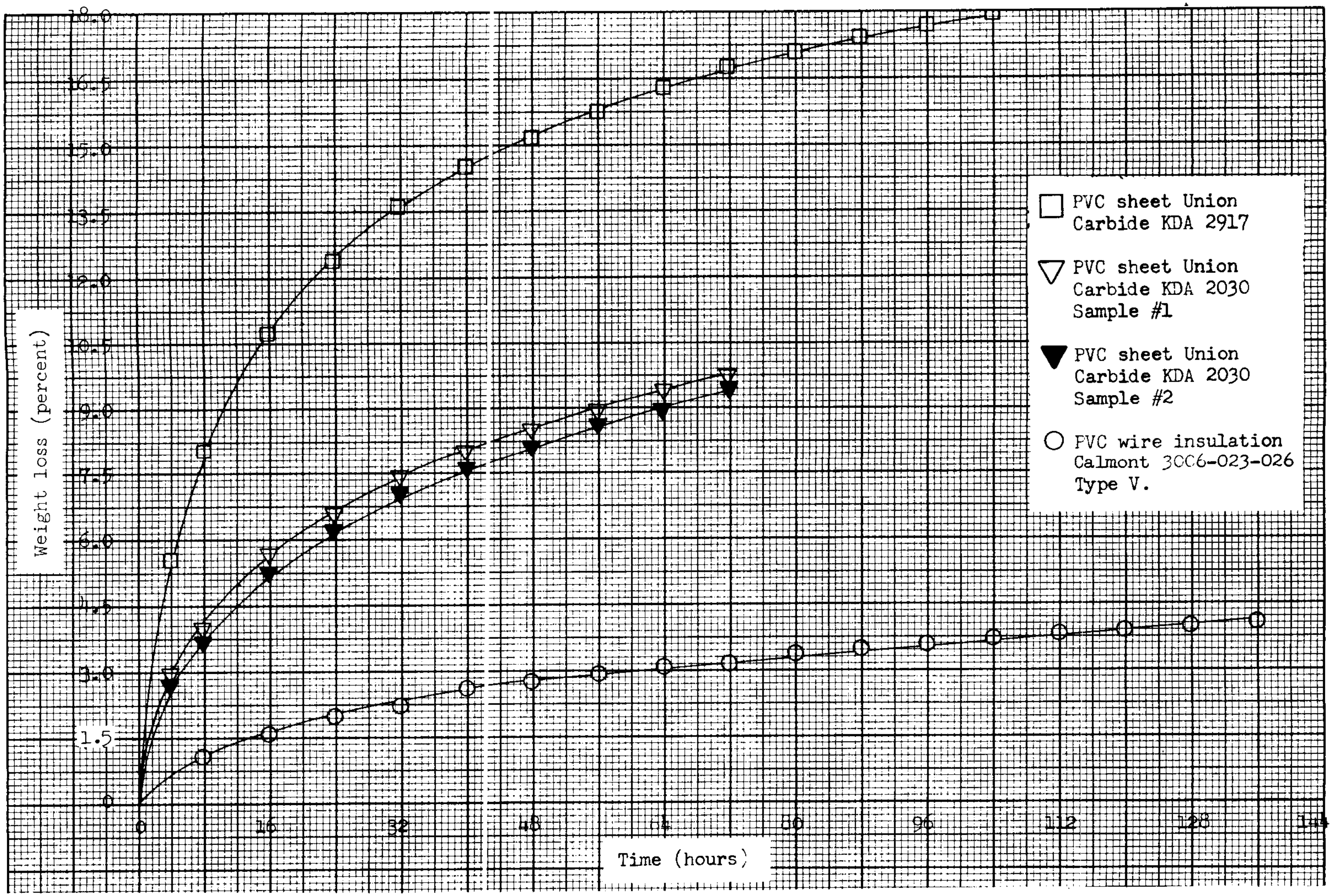

Figure 1.- Weight loss history for GT-4 PVC materials at 71° C and 10^{-4} torr.

APPENDIX A

QUALIFICATION TEST PROGRAM

Tensile Strength and Elongation Tests for Polyvinyl Chloride Film and Wire Insulation

Scope.- This test will determine the tensile strength and elongation characteristics of the PVC types to be tested. Two test environments are required:

a. Room temperature and pressure to evaluate degradation incurred during 3 psia O_2 and fungus tests.

b. After 14 days at 1×10^{-6} torr, elevated temperature (160° F) and room temperature to evaluate degradation incurred during decompressed cabin operations.

The test equipment and procedures are similar for both environments.

Sample requirements.- Film shall be $\frac{1}{2}$-inch by 12-inch swatches. Wire insulation shall be 1 foot in length. Five samples of each type shall be tested.

Test equipment.-

a. Multiple vacuum tensile tester

b. Flat face grips

c. Vacuum temperature chamber capable of operating at 160° F

Test procedure.- The material will be mounted in the grips with a distance between upper and lower grip faces of $3 \pm \frac{1}{8}$ inches. The distance between the grips shall be measured to within ± .05 inch. Chart load and elongation scales shall be set up to give a reproducible load-elongation curve. Elongation will be measured from crosshead travel. The crosshead rate of travel shall be 20 ± 1.0 inches per minute. One sample shall be used to determine load and elongation limits, then three samples shall be tested. The fifth sample will be used only in the event of questionable results on one of the three samples. Tests will be conducted at the applicable environment given above under "Scope". Temperature, pressure, and relative humidity shall be recorded. Strength and elongation shall be calculated per Federal Standard J-C-98, Methods 3021 and 3031, respectively.

Acceptance criteria.- Degradation of greater than 30 percent following vacuum/elevated temperature, 3 psia O_2, or fungus tests, shall be cause for rejection.

Special Waiver.- In view of the short time available before GT-4 launch, each material will be tested after a 4-day exposure per paragraph b under "Scope" on the preceding page. Depending on results obtained, the test will be rerun for the full 14 days at a later time.

Offgassed Products and Weight Loss in 3-psia Oxygen for Polyvinyl Chloride Film and Wire Insulation

Purpose.- To establish a procedure for sample collection and analysis of outgassing products from Polyvinyl Chloride (PVC) film and wire insulation under controlled environmental conditions.

Documents and forms.-

a. This procedure will be the document authorizing this study, and will govern the responsibilities and duties of all sections and personnel concerned.

b. MSC Form 42, Standards Laboratory Calibration Request, will be initiated by IESD Standards and Quality Assurance Branch, Analytical Instrumentation Calibration Laboratory.

c. Results of the study will be reported by the Analytical Instrumentation Calibration Laboratory in a memorandum or paper to be prepared upon completion of the study.

Scope.- This procedure outlines the responsibilities of IESD Standards and Quality Assurance Branch sections for the following:

a. Establishment and control of environmental conditions.

b. Sample preparation.

c. Sampling methods, frequency and handling.

d. Sample analysis methods, and data recording and reporting procedures.

Sample requirements.- Analytical Instrumentation Calibration Laboratory shall prepare the samples. PVC film or wire insulation, approximately 1 square yard, shall be cut, cleaned, and weighed. Wire insulation shall be stripped from conductor. Dimensions and weight shall be recorded.

Test equipment.-

a. Bethlehem Test Chamber with required instrumentation

b. Mettler precision balance

c. Vacuum pump

d. Cold trap

e. Beckman IR-9 infrared spectrometer

f. Consolidated electrodynamics 21-103C mass spectrometer.

g. Barber Colemen 5000 series gas chromatograph

Test procedures.- Prior to the start of sample testing, the chamber shall be purged with oxygen several times, pulled down to 3 psia ± .2 psia at room temperature, and a sample shall be trapped for analysis to determine chamber cleanliness.

Environmental Test and Evaluation Section of IESD Standards and Quality Assurance Branch shall be responsible for placing the sample (refer to above paragraph on "Sample requirements" for sample source and preparation) in the Bethlehem Test Chamber, purging the chamber with oxygen, establishing environmental conditions in the chamber, and maintaining these conditions throughout the test period of 14 days. This section shall provide all personnel and equipment to operate the test chamber and accomplish the above.

The sample shall be placed in the chamber, and the chamber evacuated and purged with oxygen, breathing grade, MIL-0-21749, for 1 hour at a flow rate of 1 liter per minute.

After purging, the following conditions shall be established in the test chamber:

a. 3 psia ± .2 psia of MIL-0-21749 oxygen.

b. 120° ± 2° F.

c. Relative humidity - nil

These conditions shall be maintained for the duration of the test which will be 14 days. Environmental Test and Evaluation Section shall provide necessary manifold, gages, valves, regulators, and other equipment necessary to maintain test conditions. Provisions shall be made for changing oxygen cylinders without affecting pressure within the chamber or flow through the sample-collecting system.

This test will require monitoring of chamber and controls continuously for 14 days. Chart records of temperature and pressure will be made. All charts will be forwarded to Analytical Instrumentation Calibration Laboratory upon completion of the test for inclusion in records and final report.

After weighing, the sample shall be placed in the chamber. Personnel handling the sample after cleaning shall wear rubber or polyethylene gloves to avoid contamination. Placing the sample in the chamber shall be coordinated with Environmental Test and Evaluation Section in order that purging of the chamber may start immediately after the sample is in place.

Analytical Instrumentation Calibration Laboratory shall furnish the sample-collecting system (fig. 2).

The sample will be collected by passing gases from the test chamber through a cold trap. The cold trap will consist of approximately three loops of $\frac{1}{4}$-inch stainless steel tubing. The loops will be approximately 3 inches in diameter, and will be immersed in liquid nitrogen during the sampling periods. The cold trap will be equipped with two toggle valves as shown in figure 2. Another toggle valve shall be installed directly on the test chamber. This will permit closing off the line to the chamber and sealing the cold trap for removal to the laboratory for analysis.

The sample collected shall be recovered and analyzed at the end of 2 hours, 4 days, and 14 days. To recover the trapped sample, toggle valves 1, 2, and 3 (fig. 2) shall be closed, the cold trap removed, and taken to the Analytical Instrumentation Calibration Laboratory. The cold trap shall be heated by immersion in boiling water to vaporize all trapped compounds. The vapors shall be collected in an evacuated cell for infrared spectrographic analysis.

Sample weight shall be taken following completion of the 14-day test. All effort shall be made to preclude weight gain due to atmospheric moisture pick-up.

The sample shall be cut into five $\frac{1}{2}$-inch by 12-inch swatches, and shall be subjected to the tensile and elongation tests.

The method of sample analysis snall be the infrared spectrophotometer. A mass spectrometer and gas chromatograph will be available, and may be used to verify or assist in interpretation of infrared spectrograms.

Data records will consist of test chamber logs and infrared spectrograms. Original copies of these records will be retained and filed by Analytical Instrumentation Calibration Laboratory.

A report will be prepared by the Analytical Instrumentation Calibration Laboratory. The report will consist of copies of the records mentioned above, and a brief outline of the test results giving the outgassing components found, and their concentrations, and the weight of the sample before and after test.

Acceptance criteria.-

a. The results of the 2-hour, 4-day, and 14-day infrared spectrograms will be submitted to the CSD Environmental Physiology Branch for analysis of potentially toxic products.

b. Degradation of physical properties (tensile and elongation) greater than 30 percent following 14-day exposure to 3 psia O_2 shall be cause for rejection.

Special waiver.- In view of the short time available before GT-4 launch, and since materials cannot be tested concurrently, each material will be subjected to the 4-day test only in order to evaluate its capability for the GT-4 mission. Depending on results obtained, the test will be rerun for the full 14 days at a later time.

Vacuum Volatility

Scope.- This test will determine the weight loss of Polyvinyl Chloride film and wire insulation due to outgassing under vacuum conditions. The results of this test will determine if significant amounts of condensate would be offgassed in spacecraft and deposited on optical and other critical surfaces.

Sample requirements.- Samples weighing 1 to 2 grams, weighed to an accuracy of ± .001 grams, will be subjected to the weight loss tests. Wire insulation shall be stripped from the conductor. Samples will be cleaned with ethyl alcohol. Prior to testing, the samples will have their weight and dimensions recorded. Three samples of each material will be tested.

Test Equipment.-

a. Recording balance with very-high-vacuum system. Ultimate pressure 1 or 2 $\times 10^{-7}$ torr. Sensitivity 0.1 mg.

b. Infrared heaters

c. Millivolt potentiometer

d. Microscope slide

e. High-vacuum chamber

f. Automatic temperature controller for infrared heaters

Test procedure.- Test specimen will be coiled and suspended from one arm of the recording balance. An iron constantan thermocouple insulated with 2 or 3 mils of Teflon will be inserted in the coil and lead out to a millivolt potentiometer.

A clean microscope slide will be weighed, the weight recorded, and then placed in the bottom of the sample tube of the balance. The system and sample will be closed to the atmosphere, and pumped down to lowest attainable vacuum at room temperature. When the weight recording mechanism of the balance shows a flat line (no weight loss) at room temperature, infrared heaters will be activated to give a temperature of 160° F on the sample.

Heat will be applied and continuous weight loss rate measurements shall be recorded. If the slope of the curve obtained at 160° F is less than 0.0025 percent per hour the test will be terminated and the sample allowed to cool to room temperature.

If the slope is greater than 0.0025 percent per hour, the temperature will be reduced to 25° F steps, and the highest temperature determined at which the sample exhibits a stable rate less than 0.0025 percent per hour.

Following completion of the rate measurements, the sample shall be permitted to cool to room temperature. Air shall be admitted to the sample chamber, and weight gain during the first 30 minutes of air exposure shall be noted.

The glass slide shall be removed from the system and weighed. It shall also be visually examined for evidence of condensate.

Total weight loss for the sample (excluding weight gain on admission of air) shall be recorded. Any weight gain noted shall be recorded.

Weight loss of the material in percent shall be plotted against the time in hours.

Acceptance limits.- PVC material is considered acceptable if the stable rate of weight loss is not greater than 0.0025 percent per hour at 160° F and the total equilibrium weight loss is not greater than 2 percent in the first 24 hours, based on the original weight of the material.

If the material does not meet these limits, attempts will be made to pretreat in vacuum to reduce weight losses to acceptable levels.

Fungus Test for Equipment Containing Polyvinyl Chloride Film and Wire Insulation

Scope.- This test specification is designed to insure satisfactory operation of equipment containing Polyvinyl Chloride (PVC) during spacecraft operations. It is designed only to be applicable where equipment is stored in moderate humidity and temperature conditions. It is designed for equipment which is placed in direct contact with the skin during mission operations. It is designed to qualify equipment for missions of up to and including 21 days.

Sample requirements.- Five swatches $\frac{1}{2}$ inch by 12 inches, five 1-foot wire samples, and five examples of each equipment type shall be tested.

Test equipment.- Incubator capable of 40° C and 95 percent ± 5 percent relative humidity.

Procedure.- Mixed flora of bacteria and fungi obtained from the axilla and groin are prepared under standard conditions and applied to the equipment to be tested. After this application, the swatches and the equipment, including applicable external connections, shall be placed in a chamber maintaining an internal temperature of 37° ± 3° C and a relative humidity of 95 percent ± 5 percent and retained for 21 days. At the end of the test, the swatches and the equipment shall be visually examined in accordance with standard procedures. The equipment shall then be operated and inspected as outlined in MIL-E-5272C (ASG) amendment 1, paragraph 3.8, dated January 20, 1960. The swatches shall be subjected to the Tensile and Elongation Test. This test shall be repeated for all swatches and devices using mixed cultures obtained from the crew members involved, or if not available, an equivalent match group of adult males.

Acceptance criteria.- The equipment shall be capable of performing required functional tests with no degradation of performance after the 21-day exposure. Any fungus build-up on the PVC material tested shall not present a bacteriological hazard to the astronaut either during the longest mission or during the post-landing period; nor shall such build-up interfere with required suit circuit oxygen flow, elimination of normal body wastes, or present a mobility hazard to the suited astronaut.

Degradation of greater than 30 percent in physical properties (tensile and elongation) shall be cause for rejection.

Special waiver.- In view of the short time available before GT-4 launch, each material will be subjected to a 4-day test only. Depending on results obtained, the test will be rerun for the full 14 days at a later time.

Odor Test and CO Measurement for Polyvinyl Chloride Film and Wire Insulation

Purpose.- The odor and CO test is designed as a nonmetallic materials screening test of spacecraft materials located in the spacecraft habitable areas.

Selection of test panel.- The contractor shall establish a pool of qualified test subjects from which a test panel of at least five members shall be selected for odor testing evaluation. Members of the panel shall be male, and qualified in the following manner: Each candidate shall be capable of discriminating between seven basic odors when presented with a freshly prepared dilution of each according to the following schedule.

Primary odor	Standard compound	Standard ppm concentration	Experimental dilution (In H_2O)
Ethereal	1,2-Dichloroethane	800 v/v	0.4 ml → 500 ml
Camphoraceous	1,8-Cineole	10 v/v	5μ l → 500 ml
Musky	15-Hydroxypentadecanoic acid lactone	1 w/v	1 mgm → 1000 ml (warm water to 70° F to melt)
Floral	D,L-8 Phenylethylmethyl ethyl carbinol	300 v/v	0.075 ml → 250 ml
Minty	d,L-Menthone	6 v/v	2μ l → 333 ml
Pungent	Formic acid	50 000 v/v (5 percent)	25 ml of 90 percent solution → 500 ml
Putrid	Methyl Disulfide	0.1 v/v	1μ l → 500 ml 25 ml of this → 500 ml

Odor criteria.-

a. Odor detection: A test panel of at least five subjects shall be used to sense the odor of the test samples. The vapor sample shall be presented at an initial dilution of 1:100 by gas volume. Dilution shall be by means of the same oxygen used to prepare the test sample. A 20-cc vapor sample shall be introduced in approximately 3 seconds by means of a hypodermic syringe, into a mask of odorless, flexible material applied to the subject's face as per figure 3. The subject shall inhale as the sample is introduced. A minimum of ten individual test trials will be performed for each given sample. In the event that the test panel consists of five to nine members, each member shall evaluate the test sample twice. In the event that the test panel consists of ten or more members, each member shall evaluate each sample once.

b. Evaluation: Each test panel member shall evaluate the odor of the sample according to the following verbal scale.

	Rating (for test conductor only)
Undetectable	Zero
Barely detectable	One
Easily detectable	Two
Strong and/or irritating	Four

The test conductor shall calculate an arithmetic average for the ratings of the total test. A mean score of 2.0 or less signifies an acceptable odor screening. Scores in excess of 2.0 denote unacceptable materials based upon odor. Odorless vapors may be introduced as a check on the performance of the test group at the discretion of the test conductor.

In addition, the material will be unacceptable if a concentration in excess of 25 ppm CO is detected. This would correspond to a concentration of .25 ppm in the spacecraft.

Test equipment. -

a. Test chamber: A pyrex glass container of known volume (about 2 liters) equipped with (1) a gas-tight removal cover, (2) valve, and (3) a sampling plug. A laboratory desiccator may be utilized. All sealants, valve packings, and other nonmetallic parts shall be nonodor-producing under the test conditions.

b. Heat source: The heat source shall be a means of providing a constant temperature of 200° ± 5° F, and shall be external to the test chamber. The oven temperature shall be monitored and controlled.

c. Oxygen supply: A source of low pressure MIL-0-27210, Type 1, oxygen with suitable equipment for reading pressure and for purging the test chamber as required.

d. Sample transfer system: The gas sample measuring and transfer system shall consist of glass syringes equipped with sampling tubes or needles.

e. Olfactometer shall consist of a mask of odorless, flexible material which can be applied to the subject's face as shown in figure 3.

Sample preparation.-

a. Candidate materials will be prepared or processed by the normal production methods.

b. Bulk materials will be processed into $\frac{1}{2}$-inch by $\frac{1}{8}$-inch strips with sufficient length to make up a 10 ± 0.5 g sample, or 5 ± 0.25 g sample per liter of test chamber volume.

c. Film and fabric materials will be tested in their "as purchased" condition, with sufficient area to make up a 10 ± 0.5 g sample.

d. Tapes, primers, coating materials, and adhesives will be applied to a chemically clean .030-inch aluminum substrate panel, in a thickness equal to production practice. The total coated panel area will be 1 square foot. The panels may be cut into smaller squares for convenience in exposure in the test chamber. The panels are to be coated on one side only.

e. Liquids, greases, oils, coolants, et cetera will be tested as 10 ± 0.5 g samples contained in aluminum weighing dishes of approximately 2-inch diameter.

Test conditions.-

a. The atmosphere in the test chamber at the start of the test exposure shall be MIL-O-27210, Type 1, oxygen at 5 ± 0.3 psia.

b. The materials being tested shall be heated at 200° ± 5° F for 3 hours.

c. All equipment shall be clean and free of extraneous odors. Metal parts shall be chemically cleaned in accordance with commonly accepted shop practice. Glass parts shall be cleaned in a standard sulfuric acid-dichromate solution, followed by a thorough hot water rinse, distilled water rinse, and oven-dried at a minimum temperature of 200° F.

d. The test system shall be free of leaks.

e. Individual test chambers, as defined on the preceding page under "Test Equipment", shall be used for each material sample.

f. Actual odor evaluation shall be carried out within 4 hours of the conclusion of the thermal exposure.

Test Procedure.-

a. Material samples shall be weighed to the nearest 0.1g prior to conditioning for a minimum of 24 hours in a standard desiccator containing MIL-O-27210, Type 1, oxygen at one atmosphere.

b. Material samples will be transferred to the test chamber, which has been previously cleaned and charged with oxygen during inert storage. Transfer will be made with as little exposure to normal atmosphere as possible.

c. The test chamber will be evacuated to at least 1 mm. The chamber will then be charged with 5 ± 0.3 psia oxygen.

d. A leakage check will be made by observing pressure increase in the test chamber. There shall be no measurable pressure rise in one-half hour using an instrument sensitive to 1.0 torr.

e. The test chamber will be exposed to a temperature of $200 \pm 5°$ F for 3 hours allowing time for initial warmup.

f. Following thermal exposure, the chamber will be returned to ambient room temperature.

g. Test chamber pressure will be measured and recorded.

h. Observation of distillate residues on interior chamber walls will be made and recorded.

i. The test chamber will be pressurized to one atmosphere with oxygen, and sampling plug installed. Chamber dilution will be one part contaminated oxygen and two parts fresh oxygen for calculation purposes.

j. Known volumes of sample atmosphere will be extracted from the test chamber and diluted 1:33 by gas volume with fresh oxygen. 20 cc of diluted gas shall be introduced, by a glass syringe, into the face mask in 30 seconds (see fig. 3). If larger than 2 liter desiccators are used, the additional volume must be taken into consideration, or utilize more sample according to Sample Preparation, paragraph b.

k. If no odor is detectable, test the gas samples at 1:10 and 1:3 dilution.

l. The sample will be tested for (CO) carbon monoxide using a suitable detector such as the Kitagawa Precision Gas Detector Model 15-3000.

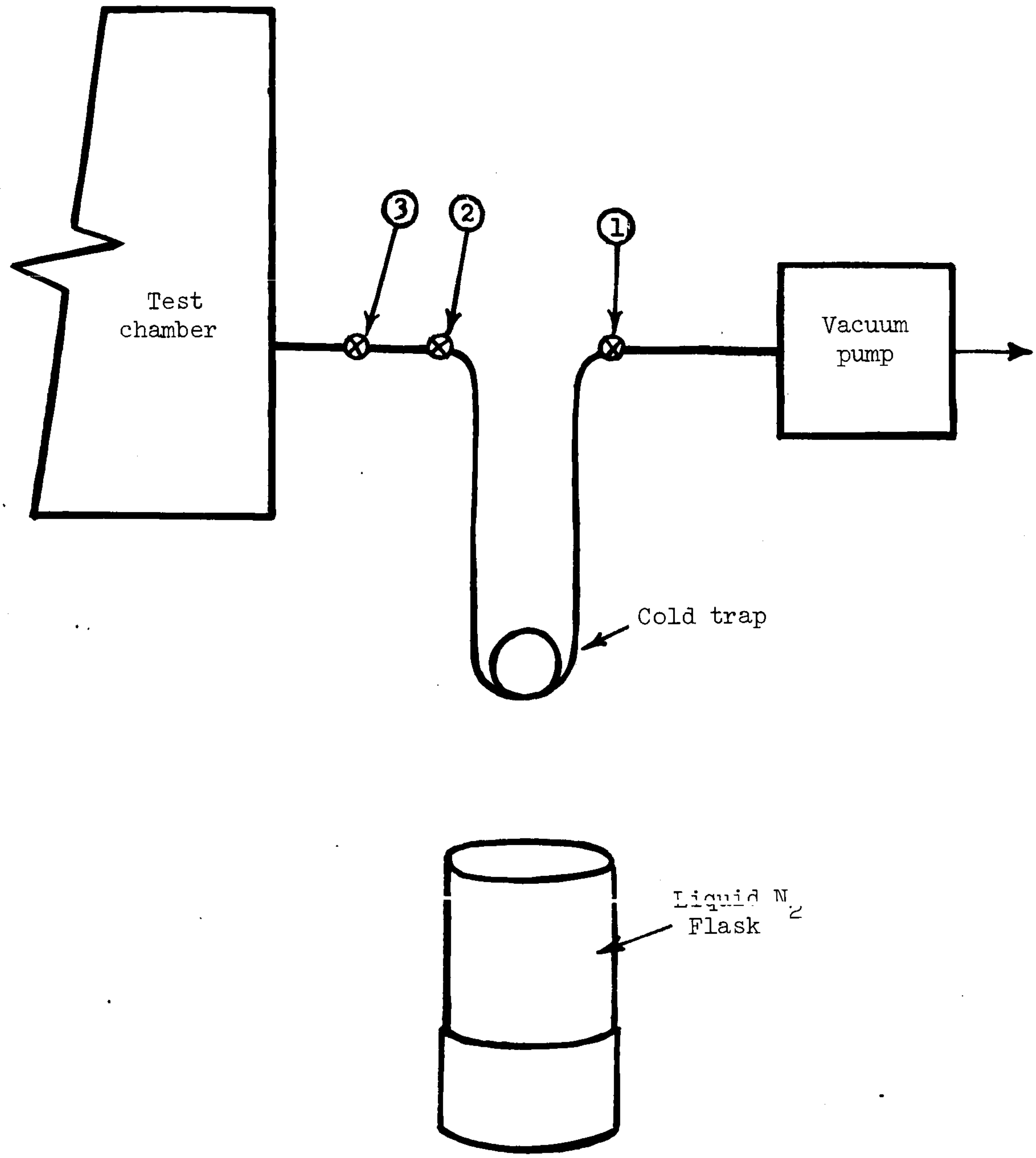

Figure 2.- Sample collecting system

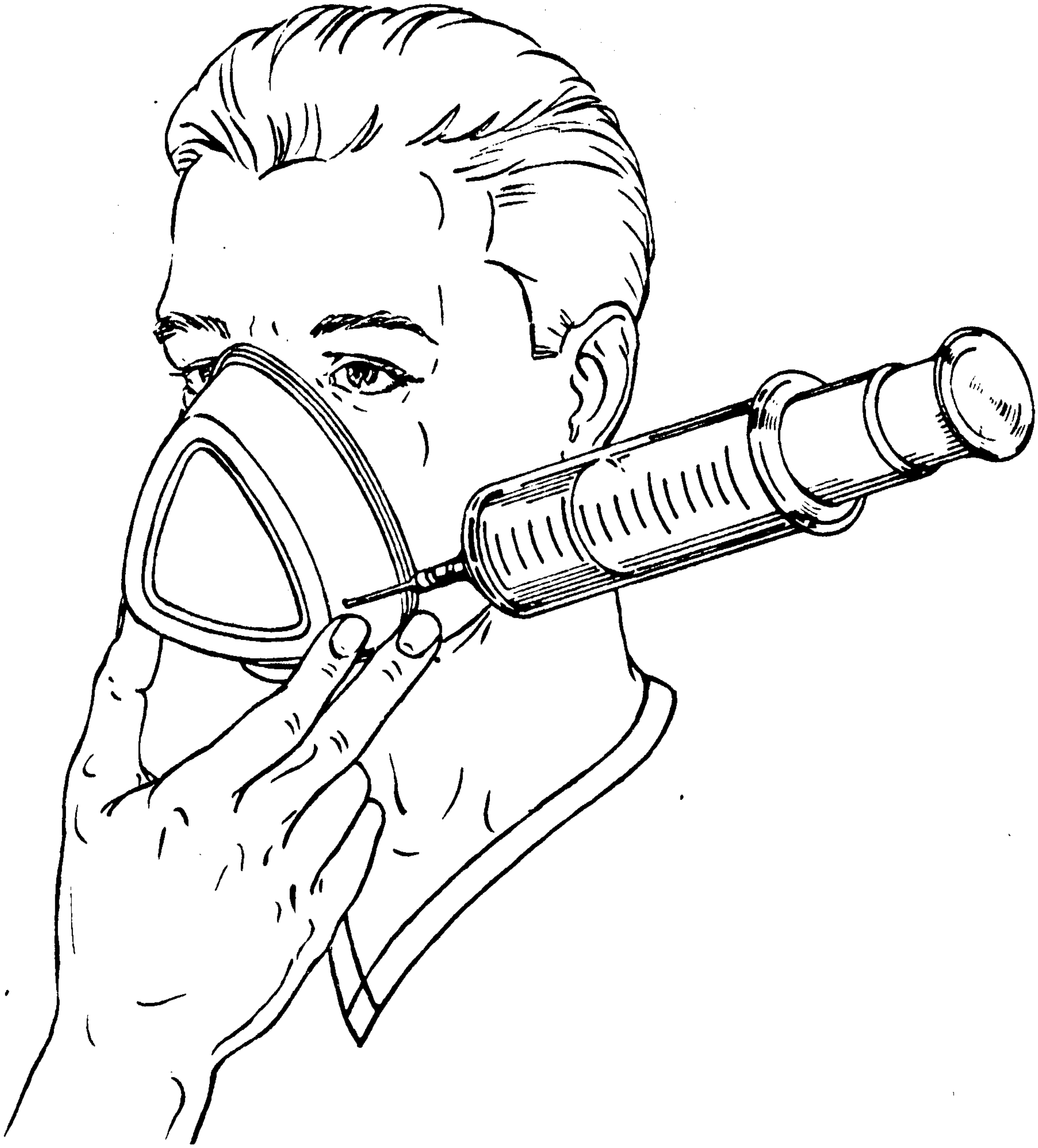

Figure 3.- Introduction of gas, using glass syringe

APPENDIX B

CONTRIBUTORY SOURCES

The following list of individuals and departments constitutes the main sources of the information contained within this publication, as gleaned from miscellaneous memoranda exchanged and submitted for consideration:

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